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FORM I		90 (Modified)  U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE  ATTORNEY'S DOCKET NUMBER
ł	Tŀ	RANSMITTAL LETTER TO THE UNITED STATES  4296
		DESIGNATED/ELECTED OFFICE (DO/EO/US)  U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR
		CONCERNING A FILING UNDER 35 U.S.C. 371 09/646978
INTER PC:	NAT	IONAL APPLICATION NO. INTERNATIONAL FILING DATE PRIORITY DATE CLAIMED January 28, 1999
		INVENTION
L	IGH	T-EMITTING MATERIAL AND PRODUCING METHOD THEREOF
APPL	ICAN'	T(S) FOR DO/EO/US 1) Qinglong HAO, 2) Pengcheng LI, 3) Qian XIU,
		4) Atsushi OGURA and 5) Jingfeng GAO
Appli	cant h	herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information.
1.	X	This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
2.		This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
3.		This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay
4.		examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).  A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5.	<u>∑</u>	A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
	-	a.  is transmitted herewith (required only if not transmitted by the International Bureau).
		b. 🖾 has been transmitted by the International Bureau.
me. me		c. is not required, as the application was filed in the United States Receiving Office (RO/US).
<u>1</u> 6.	X	A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7.		A copy of the International Search Report (PCT/ISA/210).
<b>8</b> .		Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
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		c. $\square$ have not been made; however, the time limit for making such amendments has NOT expired.
#: j=4		d.  have not been made and will not be made.
= <b>9</b> .		A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
TO.	X	An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
LI.		A copy of the International Preliminary Examination Report (PCT/IPEA/409).
9		A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).
	ems 1	13 to 18 below concern document(s) or information included:
13.		An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14.	Ø	An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15.	M	A FIRST preliminary amendment.
		A SECOND or SUBSEQUENT preliminary amendment.
16.		A substitute specification.
17.		A change of power of attorney and/or address letter.
18.	Ø	Certificate of Mailing by Express Mail
19.	×	Other items or information:
		THE FEE CACULATION MUST BE BASED ON THE CLAIMS AS AMENDED AND ADDED IN THE ATTACHED PRELIMINARY AMENDMENT.
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### IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE MATTER OF:

APPLICANT: HAO et al

ORDER/DOCKET NO. 4296

FOR: LIGHT-EMITTING MATERIAL AND PRODUCING METHOD THEREOF

### PRELIMINARY AMENDMENT

Hon. Commissioner of Patents and Trademarks Washington, DC 20231

SIR:

Please amend the application as follows.

#### IN THE CLAIMS

Please amend claim 8 as follows:

Claim 8, line 2, delete "or 7"

Please add new claim 9 as follows:

- - 9. A producing method of a light-emitting material according to claim 7, wherein in said step (2), reduction is carried out using carbon powder. - -

## **REMARKS**

A Preliminary Amendment has been made to correct multiple dependent claim 8.

It is respectfully requested that the above amendment be entered before calculation of the filing fee and before examination by the Examiner.

Respectfully submitted,

Eugene Lieberstein Registration No. 24645

ga0721ha.amd

Anderson, Kill & Olick, P.C. 1251 Avenue of the Americas New York, NY 10020-1182 212/278-1000

AE. HAO et Al ORL 4296 09/646978 529 Rec'd PCT/PTO 25 SEP 2000

#### **SPECIFICATION**

LIGHT-EMITTING MATERIAL AND PRODUCING METHOD THEREOF

#### TECHNICAL FIELD

The present invention relates to a light-emitting material and a producing method thereof, and more particularly, to an inorganic light-emitting material using a rare-earth element as an exciting agent and having an afterglow time and a producing method thereof.

#### BACKGROUND TECHNIQUE

A light-emitting material is utilized is mixed in ink or paint to make light emitting paint, and is utilized on a safe sign and a clock board. Conventionally, copper-excited zinc sulfide (ZnS: Cu) was widely used as the light-emitting material. ZnS: Cu has high light-emitting efficiency in the light-emitting spectral region, but its brightness is lowered extremely rapidly, and the visible afterglow time is as short as 20 to 30 minutes. When the ZnS: Cu is exposed to ultraviolet rays in areas exposed to moisture, decomposition and degeneration are generated and the body color of the material darkens. Therefore, there are constraints for using ZnS: Cu outside, and optimal material as a substitute for ZnS: Cu had long been required.

In CN1053807A, a light-emitting material (m(Sr<sub>1-x</sub>Eu) O·nAl<sub>2</sub>O<sub>3</sub>-yB<sub>2</sub>O<sub>3</sub>) having long afterglow ability and is laid open. In the above general formula,  $1 \le m \le 5$ ,  $1 \le n \le 8$ ,  $0.005 \le y \le 0.35$  and  $0.001 \le x \le 0.1$ . The afterglow time of this light-emitting material is in a range from 10 to 20 hours.

In USP5,376,303, phosphor having long afterglow ability comprises a compound (MO a  $(Al_{1-b}B_b)_{2}O_3$ : cR).

In this general formula,  $0.5 \le a \le 10.0$ ,  $0.0001 \le b \le 0.5$ ,  $0.0001 \le c \le 0.2$ , MO is a at least one compound selected from a group consisting of MgO, CaO, SrO and ZuO. R consists Eu and at least one additive rare-earth element selected from a

group consisting of Pr, Nd, Dy and Tm.

In the above-mentioned patent, some phosphors such as SrO. 2.10  $(A_{10.952}B_{0.048})$   $_2O_3$ : 0.005Eu, 0.020Dy (which will be referred to as "A" hereinafter), and  $SrO._{1.025}$   $(Al_{0.976}B_{0.024})_2O_3$ : 0.005Eu, 0.015 Dy (which will be referred to as "B" hereinafter) were prepared, and residual light-emitting time and brightness of these phosphors were evaluated in view of afterglow time constant (n) and relative brightness (ZnS: Cu, Cl were evaluated). A measuring result shows that the afterglow time constants of A and B phosphors and ZnS: Cu, Cl are 0.94, 0.86 and 1.26, respectively, and phosphorus brightness after 10 seconds are 144, 220 and 100, respectively, and phosphorus brightness after 20 seconds are 934, 1320 and 100, respectively.

It is found that these materials are clearly improved as compared with ZnS: Cu, Cl, but they are not yet in practical use.

Thereupon, the present inventors conducted various researches for producing light-emitting material using rare-earth element Eu as the light-emitting material. Based on the researches, the present inventors found that a light-emitting material having a new crystallization structure was obtained by adding an appropriate amount of B and an additive exciting agent. This light-emitting material has a desired long afterglow time and high brightness.

Therefore, a first object of the present invention is to provide a light-emitting material having a long afterglow time and high brightness.

Moreover, a second object of the present invention is to provide a producing method of the above-mentioned light-emitting material.

#### DISCLOSURE OF THE INVENTION

To achieve the objects, the present invention provides the following light-emitting material and the producing method. That is, a light-emitting material of the present invention includes a diplophase compound crystallization expressed in a general formula: (Sr, Eu, Dy) $_{0.95\pm x}$ (Al, B) $_2$ O $_3$ . $_{95}$  $_{\pm x}$ . (Sr, Eu, Dy) $_{4-x}$ (Al, B) $_{14}$ O $_{25-x}$ (x=0.01  $\sim$ 0.1).

This diplophase compound has a new crystallization structure and consists of two phases, i.e., (Sr, Eu, Dy)  $_{0.95}$   $_{\pm_x}$  (Al, B)  $_2O_3._{95\pm_x}$  and (Sr, Eu, Dy) $_{4-x}$ (Al, B)  $_{14}O_{25-x}$ . This conclusion was obtained from XRD (X-ray diffraction) analysis using a Chinese geological university material scientific divisional crystallization structure and a large amount of samples carried out in a crystallization chemical laboratory. Based on the XRD analysis, and using an optical microscope and electronic probe analytical technique, it is corroborated that the above two phases are symbiosis and have light-emitting function.

The producing method of the light-emitting material includes the following steps:

- (1) step for measuring previously pulverized raw materials, and mixing them to obtain a mixture of raw material,
- (2) step for putting the mixture into a container, heating the mixture from  $850^{\circ}$  to  $1200^{\circ}$  for three hours under a reduction condition, keeping the temperature for five to six hours, thereby obtaining a sintered body,
- (3) step for stopping the heating operation and cooling the sintered body nature down to a room temperature, and
- (4) step for pulverizing the sintered body to obtain a product.

According to the light-emitting material and the producing method of the present invention, there is a light-emitting effect that which visible long afterglow ability as compared with a similar light-emitting material.

#### BEST MODE FOR CARRYING OUT THE INVENTION

In an assessment process concerning a phase of a light-emitting material of the present invention, using analytic means such as an X-ray fluorescent analysis, a plasma spectral analysis, electronic probe and X-ray photoelectron

energy spectrum, it was confirmed that a content of B element in diplophase compound is  $0.2\sim1.0$  % by weight in general, and variable ranges of contents of Eu element and Dy are from 0.5 to 3.0 % by weight and from 0.01 to 3.0 % by weight, respectively.

The element B exists in the entire crystallization structure. Moreover, the element B exists in a form of B-O tetrahedral coordination or BO<sub>3</sub> triangular coordination. The BO<sub>3</sub> Triangular coordination can substitute a portion of Al-O octahedron and this causes instability in the crystallization structure. This is an important structural feature of the light-emitting material of the present invention.

Further, the Al-O octahedron and Al-O tetrahedron concurrently exist in the diplophase compound crystallization of the present invention, and form a substantially hexagonal ring and positive ions of Sr, Eu and Dy are charged into a cavity of the ring. From the viewpoint of the entire crystallization diplophase compound, Al exists excessively and (Sr, Eu, Dy) are insufficient.

The raw materials which is used for the producing method of the light-emitting material of the present invention are  $SrCO_3$ ,  $Al_2O_3$ ,  $H_3BO_3$ ,  $Eu_2O_3$  and  $Dy_2O_3$ , of which, Eu3+ of  $Eu_2O_3$  is reduced by Eu2+ during sintering process to excite the diplophase compound and provide the same with a light-emitting function.  $Dy_2O_3$  strengthens the exciting effect of  $Eu_2O_3$  as an additive exciting agent.

The term "reduction condition" used in the present invention means to reduce the above-mentioned mixed raw material using carbon powder, or to reduce the mixed raw material using mixture gas of nitrogen and hydrogen of volume ratio of 4:1. The light-emitting material produced by the invention has faint yellow-green color. When this light-emitting material is irradiated with sunlight, a fluorescent light or the other artificial light source and excited, the main peak of the light-emitting spectrum is  $505\,\mu\text{m}$ , and shows

blue or green.

As a result of measurement of samples, it was found that the light-emitting material of the present invention showed brightness of about 8500mcd/m² after five seconds from the instant when the irradiation was stopped, and visible afterglow time was 80 hours or longer (see Table 1). As shown in Table 1, the light-emitting material produced by the method of the present invention has especially excellent visible afterglow time.

The brightness is measured by the following method. That is, 0.2g of sample is put in a plastic plate of 10mm diameter and it is irradiated for 15 minutes from a perpendicular distance of 20cm using a fluorescent light of 15w at a room temperature and under humidity of 25RH%, and brightness of each sample is measured at various time points using an luminance meter (TOPCONBM-5, Japan TOPCON Inc.).

The light-emitting material produced by the method of the present invention has apparently long afterglow time in comparison with similar other products. Therefore, this material can suitably be applied to articles or safe sign which need to be seen in the dark, for example, a fireplug of a fire extinguishing tools and material, a handrail of safe stairs, and a road.

The following embodiments are for explaining the present invention in more detail, and are for limiting the invention. [First Embodiment]

Previously pulverized 372.89g of  $SrCO_3$ , 220.32g of  $Al_2O_3$ , 12.616g of  $H_3BO_3$ , 2.42g of  $Eu_2O_3$ , and 0.157g of  $Dy_2O_3$  were measured and sufficiently mixed. The mixed raw material was put into a container and it was covered with carbon powder, and heated from  $850^{\circ}$ C to  $1200^{\circ}$ C for three hours to increase it temperature, and the temperature was maintained for six hours. Then, the mixture was naturally cooled down to a room temperature to obtain a sintered body. The obtained sintered body was pulverized into such small pieces that all the pieces could pass through 200 mesh, thereby obtaining a product.

The product obtained in this manner had initial brightness of  $3850\text{mcd/m}^2$  for 30 seconds and afterglow time was 85 hours. In the obtained produce, a value of x in the general formula was 0.01.

#### [Second Embodiment]

Previously pulverized 409.79g of  $SrCO_3$ , 220.32g of  $Al_2O_3$ , 12.616g of  $H_3BO_3$ , 2.96g of  $Eu_2O_3$ , and 0.164g of  $Dy_2O_3$  were measured and sufficiently mixed. The mixed raw material was put into a container and it was covered with carbon powder, and heated from  $850^{\circ}C$  to  $1000^{\circ}C$  for three hours to increase it temperature, and the temperature was maintained for six hours. Then, the mixture was naturally cooled down to a room temperature to obtain a sintered body. The obtained sintered body was pulverized into such small pieces that all the pieces could pass through 200 mesh, thereby obtaining a product.

The product obtained in this manner had initial brightness of  $3990\text{mcd/m}^2$  for 30 seconds and afterglow time was 80 hours.

In the obtained produce, a value of x in the general formula was 0.01.

Table 1

Measurement of brightness  $(mcd/m^2)$  and calculation of standard deviation

deviation	Relative	standard	deviation	0.5%	1.8%	2.7%	2.3%	3.4%	2.1%	2.6%	2.48	2.1%	3.0%	2.8%	4.9%	8.3%	8.6%	12.5%	15.3%	20.5%	25.0%	33.6%	
Calculation of standard deviation	Standard	deviation		45	125	135	91	110	53	47	36	18	22	17	15	15	5.5	5.5	5.5	4.5	4.5	5.5	
Calculation	Average value			8430	7460	5044	3930	3238	2476	1810	1470	844	730	614	308	144	64	44	36	22	18	14	
	2			8500	7320	4870	3820	3110	2420	1750	1420	820	700	290	290	130	09	40	30	20	20	10	
	4			8500	7410	5170	3990	3310	2490	1830	1480	840	730	019	310	140	09	20	40	20	20	20	
No.	က			8400	7420	4930	3850	3160	2430	1780	1460	840	720	610	300	140	60	40	30	20	10	10	
Sample	7			8450	7570	5130	3960	3230	2490	1820	1470	850	740	630	310	150	70	40	40	30	20	10	
	-			8400	7380	5120	4030	3380	2550	1870	1520	870	092	630	330	160	70	20	40	20	20	20	
	Time			5 s	10 s	20 s	30 s	40 s	s 09	s 06	3min	4 min	5 min	3 min	15 min	30 min	60 min	90 min	120 min	180 min	240 min	360 min	

#### WHAT IS CLAIMED IS:

- 1. A light-emitting material including diplophase compound that is expressed in the following general formula:
- (Sr, Eu, Dy)  $_{0.95\pm x}$  (Al, B)  $_20_{3.95\pm x}$  (Sr, Eu, Dy)  $_{4-x}$  (Al, B)  $_{14}O_{25-x}$  (in the formula, x=0.01 to 0.1, a content of B element is 0.2 to 1.0 % by weight, a content of Eu is 0.5 to 3.0 % by weight and a content of Dy is 0.1 to 3.0 % by weight).
- 2. A light-emitting material according to claim 1, wherein said diplophase compound comprises symbiotical phase (Sr, Eu, Dy)  $_{0.95~\pm x}$  (Al, B)  $_{2}O_{3.95\pm}$  from (Sr, Eu, Dy)  $_{4-x}$  (Al, B)  $_{14}O_{25-x}$ .
- 3. A light-emitting material according to claim 1, wherein Al-O tetrahedron and Al-O octahedron concurrently exist in said diplophase compound.
- 4. A light-emitting material according to claim 1, wherein  $BO_3$  triangular arrangement substitute a part of Al-O octahedron in said diplophase compound.
- 5. A light-emitting material according to claim 1, wherein boron exists entirely in said diplophase compound crystalline.
- A producing method of a light-emitting material of claim1, comprising
- step for measuring previously pulverized raw materials,
   and mixing them to obtain a mixture of raw material,
- (2) step for putting the mixture into a container, heating the mixture from  $850^{\circ}$  to  $1200^{\circ}$  for three hours under a reduction condition, keeping the temperature for five to six hours, thereby obtaining a sintered body,
- (3) step for stopping the heating operation and cooling the sintered body nature down to a room temperature, and
- (4) step for pulverizing the sintered body to obtain a product.
- 7. A producing method of a light-emitting material according to claim 6, wherein said previously pulverized raw materials are  $SrCO_3$ ,  $Al_2O_3$ ,  $H_3BO_3$ ,  $Eu_2O_3$  and  $Dy_2O_3$ .
- 8. A producing method of a light-emitting material according to claim 6 or 7, wherein in said step (2), reduction is carried out using carbon powder.

#### ABSTRACT OF THE DISCLOSURE

A light-emitting material of the present invention includes diplophase compound that is expressed in the following general formula: (Sr, Eu, Dy)  $_{0.95\pm x}$  (Al, B)  $_{203.95\pm x}$  (Sr, Eu, Dy)  $_{\text{4-x}}$  (Al, B)  $_{14}\text{O}_{\text{25-x}}$  (in the formula, x=0.01 to 0.1, a content of B element is 0.2 to 1.0 % by weight, a content of Eu is 0.5  $\,$ to 3.0 % by weight and a content of Dy is 0.1 to 3.0 % by weight). A producing method of a light-emitting material of the present invention comprises (1) step for measuring previously pulverized raw materials, and mixing them to obtain a mixture of raw material, (2) step for putting the mixture into a container, heating the mixture from 850% to 1200% for three hours under a reduction condition, keeping the temperature for five to six hours, thereby obtaining a sintered body, (3) step for stopping the heating operation and cooling the sintered body nature down to a room temperature, and (4) step for pulverizing the sintered body to obtain a product. In the step (2), reduction is carried out using carbon powder.

# COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Includes Reference to PCT International Applications)

ATTORNEY'S DOCKET NUMBER

4296

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowlege the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

#### PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY UNDER 3	Y CLAIMED 5 USC 119
China	99 1 00285.7	January 28, 1999	☑ YES	□ №
		-	YES	□ NO
			YES	□ NO
	·		YES	□ NO
	·		☐ YES	□ NO

U.S. DEPARTMENT OF COMMERCE-Patent and Trademark Office

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowlege the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

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this application and	d transact all busir	iess in the Pate	ent and Trademark Office co	nnected therewith	. (List name and	registra-			
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I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowlege the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

	S.C. 120:	U.S. APPLICATIONS			STATUS (Check one)					
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